# RUBY IMPROVEMENT FOR LASERS - TASK I

## REPORT NO. 2

U. S. Army Signal Supply Agency; Contract No. DA 36-039-SC-89089
DA Project No. 3A-99-21-001 Task #1

## SECOND QUARTERLY PROGRESS REPORT

1 AUGUST 1962 TO 31 OCTOBER 1962
U. S. Army Signal Research & Development Laboratory
Fort Monmouth, New Jersey

**LINDE CO.**
DIVISION OF UNION CARBIDE CORPORATION

CRYSTAL PRODUCTS
EAST CHICAGO, INDIANA

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**RUBY IMPROVEMENT FOR LASERS TASK I**

**REPORT NO. 2**

**SIGNAL CORPS CONTRACT DA 36-039 SC89089**

**SIGNAL CORPS TECHNICAL REQUIREMENT NO. SCL-2101N**

**DA PROJECT NO. 3A 99-21-001-TASK #1**

**SECOND QUARTERLY PROGRESS REPORT**

**1 AUG 1962 TO 31 OCT 1962**

**OBJECT:** To investigate the most important variables in the Verneuil growth of ruby for laser application.


**PREPARED BY:** R. L. Hutcheson

**FOR:** Union Carbide Corporation
Linde Company Division
Crystal Products Department
4120 Kennedy Avenue
East Chicago, Indiana

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## PURPOSE

The aim of this program is to produce ruby for laser application by the flame fusion (Verneuil)[1] crystal growth process superior to presently available ruby. The program to accomplish this end is outlined in Table I (Revised 1 Sept 62). The work is divided into producing ten lots of ruby boules[2], consisting of three to five boules each. Each lot studies one to three variables with each subsequent lot being grown with the one best growth condition from the previously tested parameters.

The growth parameters being investigated are as follows:

1. Thermal gradients across the crystal during growth (Lots I through III)

2. Crystal growth rates (Lots I through VI)

3. Annealing cycles (Lots IV through VI)

4. The addition of fluxing agents (Lot VII)

5. Crystal axis orientation related to the growth direction (Lots VIII and IX)

Lot X is to be grown and annealed under the best conditions as selected from Lots I through IX and serves to summarize the work on this project.

The growth techniques employed for this work were available at Linde's East Chicago facilities prior to the inception of this contract and no new techniques are being employed. To the knowledge of the Linde Company this work is the first major attempt to relate growth parameters and crystal quality to laser performance. Information disclosed on the crystals will be only that information necessary to define the variables listed above.

Concurrent with this program the Linde Company is working on improved powders for the growth of ruby crystals via the flame fusion technique. The results of this internal work will be made available to this program at the earliest possible date. In conjunction with Task I of this contract, Perkin-Elmer Corporation is working on crystal evaluation under Task II. In addition some evaluation work will be done at Fort Monmouth and the Linde Company.

[1] Verneuil - U.S. Patent No. 1004505.(1911)

[2] Boule is a term commonly used to define the crystal grown by the flame fusion process. It originates from Verneuil's original work.

TABLE I

SUMMARY OF PROGRAM FOR "RUBY IMPROVEMENTS FOR LASERS"
(Revised 1 Sept 62)

| Lot No. | No. of Boule Per Lot | Thermal Gradients From Cap To Seed | Seed Quality | Seed Orientation | Fluxing Agent | Annealing Cycles | Relative Growth Rate | Scheduled Shipping Date |
|---|---|---|---|---|---|---|---|---|
| 1 | 3 | 300°C | See Note 2 | 90° | None | Normal | 3 | 7-31-62 |
| 2 | 3 | 150°C | See Note 2 | 90° | None | Normal | 3 | 7-31-62 |
| 3 | 3 | Approx. 50°C | See Note 2 | 90° | None | Normal | 3 | 8-31-62 |
| 4 | 5 | 50°C | See Note 2 | 90° | None | (1) Normal (Spec.Powder)<br>(2) Normal (Conv.Powder)<br>(3) In Place (Conv.Powder)<br>(4) Fast Cooling (Conv.Powder)<br>(5) Slow Cooling (Conv.Powder) | 2 | 10-1-62 |
| 5 | 5 | 50°C | See Note 2 | 90° | None | Same as Lot 4 | 4 | 10-31-62 |
| 6 | 5 | 50°C | See Note 2 | 90° | None | Same as Lot 4 | 1 | 11-30-62 |
| 7 | 3 | 50°C | See Note 2 | 90° | 0.1% Flux A<br>0.1% Flux B<br>0.1% Flux C | See Note 4 | See Note 5 | 12-31-62 |
| 8 | 3 | 50°C | See Note 2 | 0° | See Note 3 | See Note 4 | See Note 5 | 1-31-63 |
| 9 | 3 | 50°C | See Note 2 | 30°<br>45°<br>60° | See Note 3 | See Note 4 | See Note 5 | 2-28-63 |
| 10 | 3 | See Note 6 | See Note 2 | See Note 6 | See Note 6 | See Note 6 | See Note 6 | 4-30-63 |

NOTES:
1. The thermal gradient used is to be the best as determined from results of tests on Lots 1,2, and 3.
2. The seed quality is to be held constant throughout the growth of all boule lots and is to be fabricated from specially selected sapphire disc boule and used in sapphire rod holder.
3. Based on the results of tests conducted on Lots 1 through 7, it will be determined if a fluxing agent is to be used and if so, which one.
4. Based on the results of tests conducted on Lots 1 through 6, the best annealing cycle is to be determined and used.
5. Based on the results of tests conducted on Lots 1 through 6, the best growth rate is to be determined and used.
6. The decision as to the factors associated with the growth of the boules in Lot 10 will be based on the results of tests performed on Lots 1 through 9 and will represent the optimum growth condition as determined from the proposed Development Program.

TABLE I

## ABSTRACT

The effects of thermal gradients, growth rate and annealing cycles on the growth of ruby for lasers are discussed. The growth information on three lots of ruby is presented. Evaluation data shows ruby grown with low relative growth rate to have superior crystal quality.

## CONFERENCES

**Subject:** Future Contract Planning

**Persons Attending:** Fort Monmouth—Mr. Charles Kellington

Linde Company—Mr. B. N. Callihan
Mr. R. L. Hutcheson

**Held:** Linde Company, East Chicago, Indiana
9 Aug 62

**Object:** To make preliminary evaluations on boule lots I through III and make a decision on which thermal gradients would be used on the growth of boule lots IV through VI.

**Conslusions:**

1. Based on visual inspection of boule lots I, II, and III, all future ruby crystals grown for the contract would be grown at a 50°C thermal gradient as per lot III.

2. Lot IV will be delivered to Fort Monmouth two weeks late because of the delay in making the decision noted in item 1. (See note 1)

3. Lots V and VI of this contract will be delivered on schedule as per the original contract.

4. On or about 20 Nov 62 a meeting will be scheduled such that a decision based on the results of boule lots IV through VI can be made. This meeting will determine growth rates and annealing cycles that will be used to grow future boules for this contract. (See note 2)

**NOTES:**

1. Subsequent to this meeting it was determined that contract delivery dates could be met by changing the order of shipment on lots IV through VI. Therefore, a revised schedule was set up changing the growth parameters as per Table I. Also, it was decided to include one additional boule in each of lots IV, V, and VI to be grown with special alumina powder prepared by Linde Company, Speedway Laboratories, on a Linde Company funded program.

2. The meeting described under item 4 above is scheduled for 27 Nov 62 at Fort Monmouth.

## DISCUSSION

### Introduction:

The crystal growth process used to grow ruby for this contract is the flame fusion or Verneuil technique. The process is shown schematically in Figure One. Powder of sufficiently high purity and of proper dispensing characteristics is dropped into a high purity oxygen stream. The powder is carried by this stream into a furnace chamber heated by a post mixed oxy-hydrogen burner. The powder is dropped through this chamber to the molten cap of a seed crystal. A thermal gradient is established vertically down the seed crystal. This gradient allows solidification to take place on the seed in an orderly manner thereby establishing crystal growth. By proper control of the volume of the molten cap and the rate of solidification from this cap, a crystal of the desired size and shape can be grown.

The thermal gradient established vertically across the molten cap of the crystal is a factor in the resultant crystal quality. Lots I, II and III of this contract were grown for this purpose of studying the effect of this gradient on crystal quality as it pertains to laser action. As reported in the First Quarterly Report, these lots of ruby are grown by different proprietary modifications of the flame fusion process. The modifications allow the establishment of different thermal gradients between the cap and base of the crystal. These gradients down the boule can be related to the vertical gradient across the cap of the crystal. The growth techniques used are:

Lot I - Conventional (300°C gradient)
Lot II - Spiral (150°C gradient)
Lot III - Thermally Stabilized (50° gradient)

Lots I and II were presented in the First Quarterly Report. Pertinent data on lot III will follow.

Another process variable of significant importance as related to crystal quality is growth rate.[1] Large ruby crystals for industrial applications are grown relatively fast with the time being measured in hours instead of days. The influence of growth rate on laser properties has not been studied in detail. It is the purpose of lots III through VI of this contract to gain some relative insight as to the effect of growth rate on both crystal quality and laser action. The relative growth rates in ascending order are:

Lot VI - 1
Lot IV - 2
Lot III - 3

Pertinent data on lots III, IV and V are presented in this report and lot VI will be covered in the Third Quarterly Report.

Residual strain in ruby can have the effect on broadening the fluorescent band width and increasing the threshold of lasers.[2] Ruby, in its as grown state, cannot be successfully fabricated unless the boules are annealed or split. Ruby exhibits a natural spliting plane in the plane formed by the C-axis and growth axis. When boules are split along this plane the stresses are reduced sufficiently to allow fabrication of parts by conventional diamond machining technology. Variations in laser threshold as related to annealed ruby versus split ruby have been reported.[3] However, no studies on annealing cycles versus laser characteristics have been made. Lots IV through VI each are grown under constant conditions and then four of five boules are annealed by a different procedure. A fifth boule has been added to lots IV through VI to include the effect of a specially prepared alumina powder. As stated above, the pertinent data on lots IV and V, in reference to annealing are presented in this report.

Thermal Gradients:

Table II lists the growth parameters and relative evaluation data on the crystals supplied under this contract during the second quarter. The crystals prefixed CP-127 are Lot III crystals. Comparative photographs of lot III crystals are shown in Figure Two. The photographs consist of (a) the as grown crystal, (b) the Tyndall effect shown through a polished flat perpendicular to the C-axis of each crystal, and (c) the crystal between crossed polaroids.

Comparing the above information to lots I and II, as reported in the First Quarterly Progress Report, the following can be concluded:

1. There are significantly fewer bubbles in lot III crystals then lot I and II crystals.

2. There are significantly fewer lineage boundaries in Lot III crystals then in lot II crystals. Lot II crystals have fewer boundaries then lot I crystals.

3. The smoke or Tyndall effect (right angle scatter) is about the same for lots II and III. Lot I has seemingly less scatter.

4. The average $CR_2O_3$ content in lot III is lower than lots I or II, based on optical spectrophotometer measurements. However, the chromia analysis for lot I and lot II is subject to question as these initial readings did not obey Lambert's Law for absorption versus thickness.

Based on the above, a joint decision by a Fort Monmouth technical representative and Linde personnel was made to grow lots IV through VI via the thermally stabilized growth process.

Growth Rate:

Table II presents pertinent growth data and relative evaluation data on lots IV and V as indicated. Figure Three presents the relative photographs on lot IV and Figure Four presents the photographs for lot V.

Comparing lots III, IV and V, the following can be concluded:

1. The bubble content in lot IV is lowest with lot III being lower than lot V.

TABLE 11

CRYSTALS SHIPPED UNDER CONTRACT DURING SECOND QUARTER

| Lot No. | Boule No. | Specification | | | | | Powder | Thermal Gradient | Anneal | Growth Rate | Evaluation | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Length Inches | Dia. Inches | Wt. Grams | (1) Orientation Degrees | (2) % $Cr_2O_3$ Final | Source | °C | Cycle | Relative | (3) Bubbles | (4) Lineage | (5) Smoke | (6) Schlerin |
| III | CP-127-6 | 2 1/4 | 9/16 | 72 | 90 | .038 | Conv. | 50 | Normal | 2.9 | Medium | Light | Medium | - |
| | CP-127-26 | 2 15/16 | 5/8 | 95 | 90 | .040 | Conv. | 50 | Normal | 3.5 | Medium | Light | Medium | - |
| | CP-127-30 | 3 | 5/8 | 95 | 90 | .040 | Conv. | 40 | Normal | 3.0 | Light | Light | Medium | - |
| IV | CP-135-5 | 3 3/4 | 1/2 | 115 | 90 | .030 | Conv. | 60 | Slow | 2.0 | Light | Light | Medium | Light |
| | CP-135-13 | 3 15/16 | 9/16 | 143 | 90 | .033 | Conv. | 60 | Fast | 2.1 | Light | Medium | Light | Light |
| | CP-135-37 | 3 3/16 | 5/8 | 175 | 90 | .039 | Lab. | 50 | Normal | 2.0 | Light | Medium | Medium | Medium |
| | CP-135-43 | 3 5/16 | 11/16 | 147 | 90 | .040 | Lab. | 50 | In Place | 2.0 | Light | Light | Light | Light |
| | CP-134-6 | 3 7/8 | 5/8 | 124 | 90 | .030 | Conv. | 60 | Normal | 2.0 | Heavy | Light | Medium | Medium |
| V | CP-136-5 | 3 3/4 | 9/16 | 109 | 90 | .043 | Conv. | 50 | Fast | 3.8 | Medium | Medium | Medium | Medium |
| | CP-136-20 | 3 5/8 | 3/4 | 147 | 90 | .042 | Conv. | 50 | Slow | 3.5 | Medium | Medium | Medium | Medium |
| | CP-136-24 | 3 1/2 | 9/16 | 122 | 90 | .049 | Lab. | 50 | Normal | 4.0 | Heavy | Medium | Medium | Light |
| | CP-136-28 | 4 | 1/2 | 126 | 90 | .046 | Conv. | 50 | Normal | 4.0 | Medium | Medium | Medium | Medium |
| | CP-136-52 | 3 3/8 | 3/4 | 136 | 90 | .054 | Conv. | 40 | In Place | 4.0 | Heavy | Medium | Medium | Light |

1. Orientation angle is defined as the angle between the growth axis and the c-axis of the crystal.
2. % $Cr_2O_3$ is measured by comparing the optical density at 5600Å to the corrected white sapphire transmission at 5600Å.
3. Bubble content is graded by comparison to a standard boule in the area of the highest bubble concentration at 10X. The right angle scatter photographs in Figures Two and Three give a relative comparison.
4. Lineage is graded by comparison of the boule with standard boules between crossed polaroids. For examination of the boule, windows are polished perpendicular to the c-axis. The photographs shown in Figures Two and Three indicate the patterns obtained although contrast is lacking because of the black and white reproduction.
5. Smoke is graded by comparison of the Tyndall effect in the boule being compared to that same effect in a standard boule. The effect can be observed in the right angle scatter photographs shown in Figures Two and Three. End reflection must be neglected in the comparison of these boules.
6. Schlerin comparisons are explained in text of Second Quarterly Report of this contract.

TABLE 11

2. The lineage content in lot IV is lower than for either lots III or V.

3. Growth rate has little effect on smoke or Tyndall as comparative crystals are similar. However, crystal CP-135-43 made with the special powder has less smoke.

4. The chromia content in the as grown crystals follows the expected pattern; the lower the relative growth rate the larger the chromia burnout.

Based on the above evaluation, lot IV is superior in crystal quality to the other four lots of crystals supplied to date.

## Annealing Cycles:

Under the normal Linde Company proprietary process, sapphire and ruby crystals are grown by the flame fusion process and allowed to cool to room temperature. The crystals are then placed in an annealing oven, heated to over 1900°C and allowed to soak at this temperature for a period of time depending on the size, weight, and growth technique. Upon completion of the soak, the crystals are cooled to room temperature by a predertermined cycle. For fast cooling, the rate of cooling to room temperature has been increased by a factor of three. For slow cooling, the rate of cooling has been slowed by a factor of three.

A fourth annealing cycle being studied is in-place annealing. The crystal is grown, soaked at annealing temperature and hence cooled to room temperature without the immediate cooling of the normal process. The cooling cycle used for in-place annealing is comparable to the fast cooling cycle described above.

No comparisons can be made on the annealing cycles until laser data and strain measurements are available.

## Seeds:

The seeds used to grow crystals for this contract have been optically selected from disk boules. Verification of the optical selection technique via the Schultz-Wei X-Ray mapping technique, has shown misorientations to exist in the seeds and these misorientations carry into the crystals. Figure Five shows the Schultz-Wei X-Ray patterns of three seeds and the crystals grown with each seed as seen between crossed polaroids. The circled area on ,the seed pattern is the principle X-ray reflection. It can be observed that crystal CP-135-37 grown with seed 8-20-1 has no visible lineage at the seed end. Crystal CP-136-13 grown with seed 8-20-5 has considerable small angle misorientations at the seed end. These misorientations grow out of the crystal. However the major boundary of 2° continues vertically up the boule. This same effect is observed in crystal CP-136-16 grown with seed 8-20-6. This seed is a tricrystal of about 1/2° misorientation. The crossed polaroid photograph clearly shows both these boundaries carry up into the crystal.

The above discussion points out the need for seeds containing low lineage. All seeds used in future lots will be X-rayed prior to their use.

Growth Layers:

Using a crude Schlerin system, Mr. R. L. Barns of Bell Telephone Laboratories has shown grown bands to exist in ruby.(4) Figure Six shows a sketch of the optical system used and the type of banding observed. The photographs show two crystals, CP-135-43 which has the lightest, most uniform banding observed in the crystals shipped to date and CP-134-6 which shows a typical coarse band.

The cause and effect of these bands are not clearly understood. However, studies on separate programs are being made. For purposes of this report this banding will be observed and used as an evaluation technique.

## REFERENCES


1. Egli & Zerfoss and other articles; "Crystal Growth—Discussion of the Farady Society No. 5, 1949"—Butterworths, 1959.

2. A. L. Schawlow, "Advance in Quantum Electronics"—Columbia 1961.

3. R. L. Hutcheson, "Synthetic Ruby for Maser Application" presented to the IRE, March 1962.

4. R. L. Barns, "Imperfections in Ruby for Maser Application" presented to the AIME August 1962.

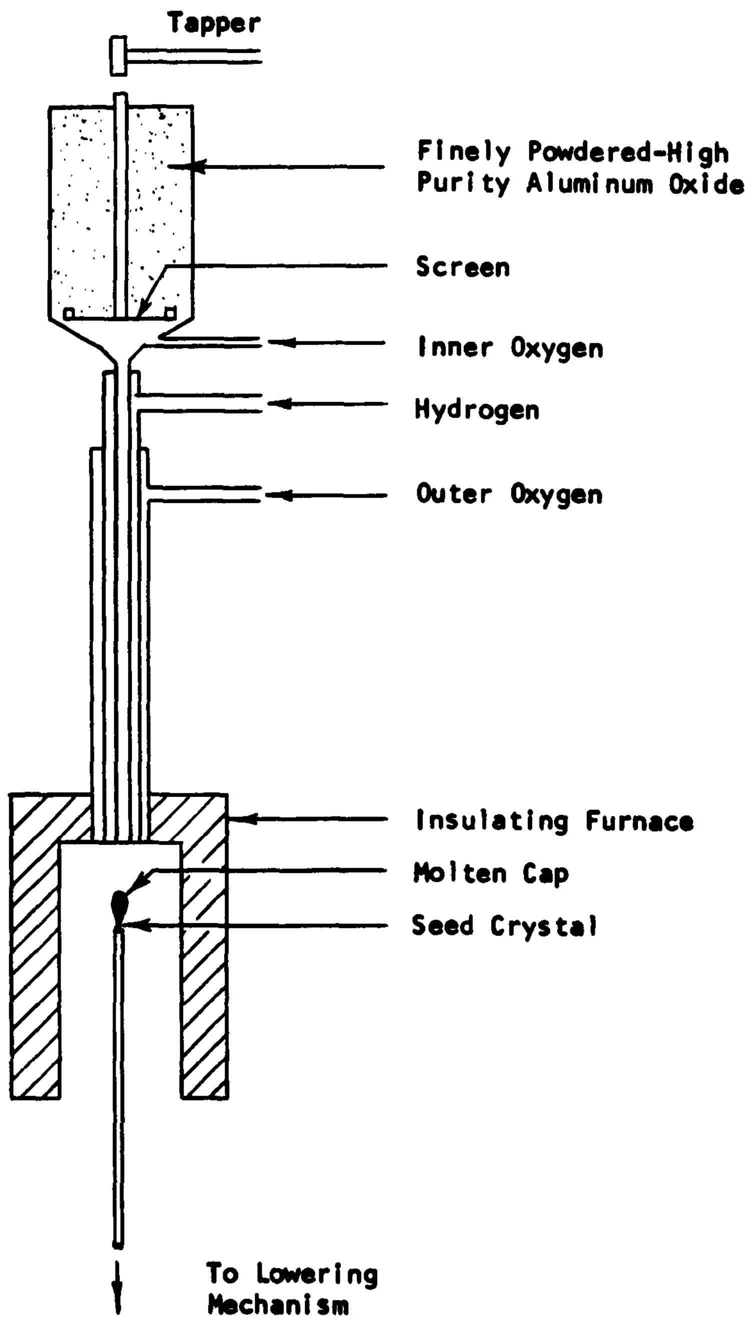
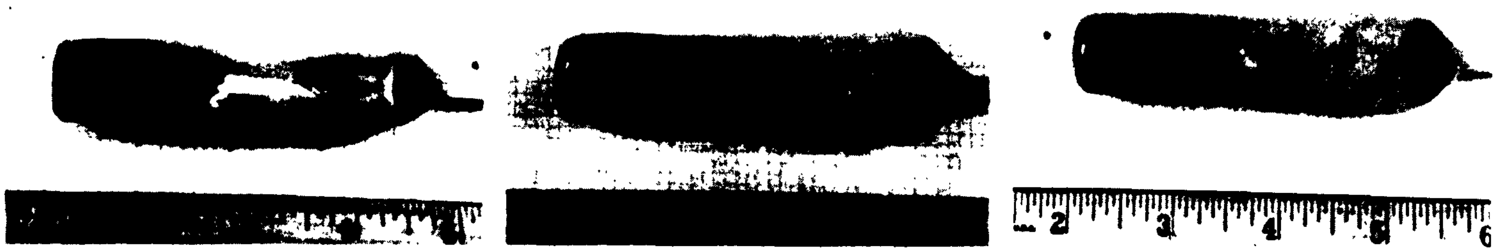


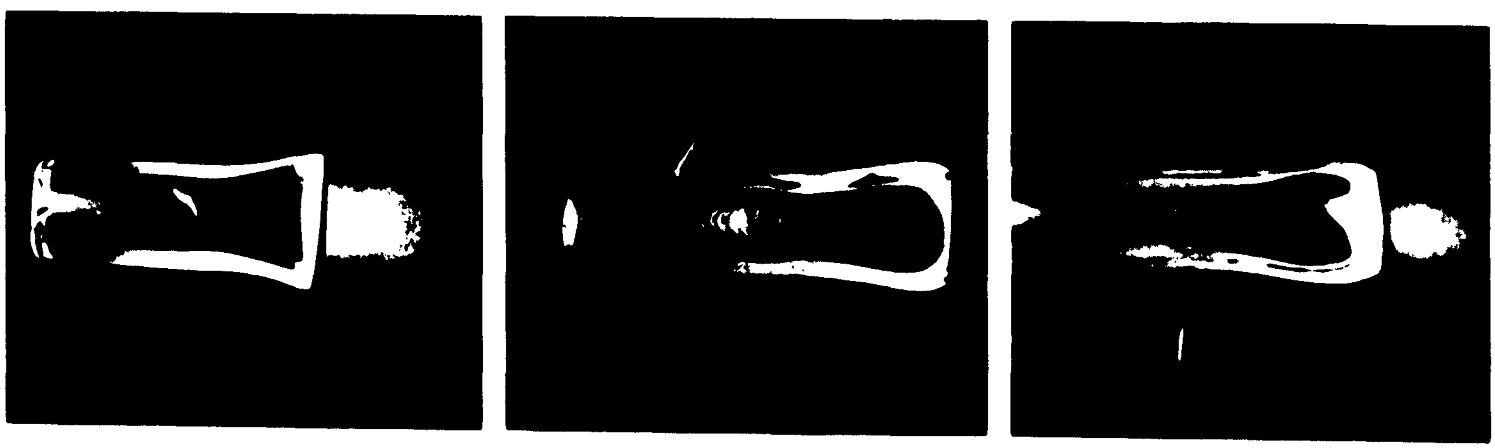

Fig. 1 Growing Sapphire by the Verneuil Technique

## PHOTOGRAPHS OF CRYSTALS SHIPPED FOR LOT III

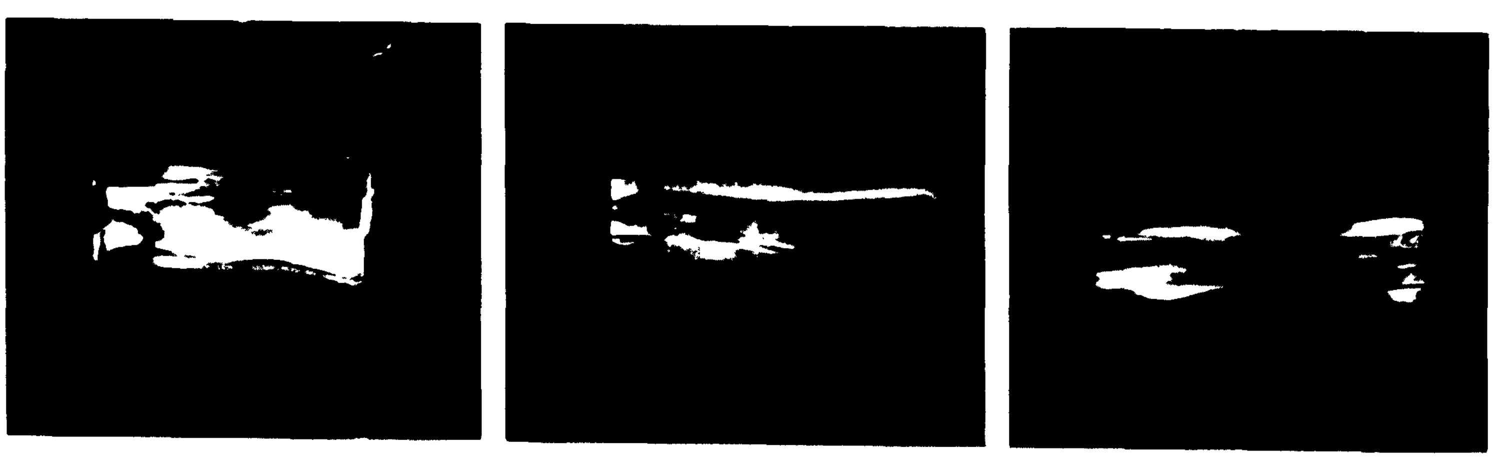
Crystal CP-127-6 | Crystal CP-127-26 | Crystal CP-127-30



A. As Grown Crystal



B. Tyndall

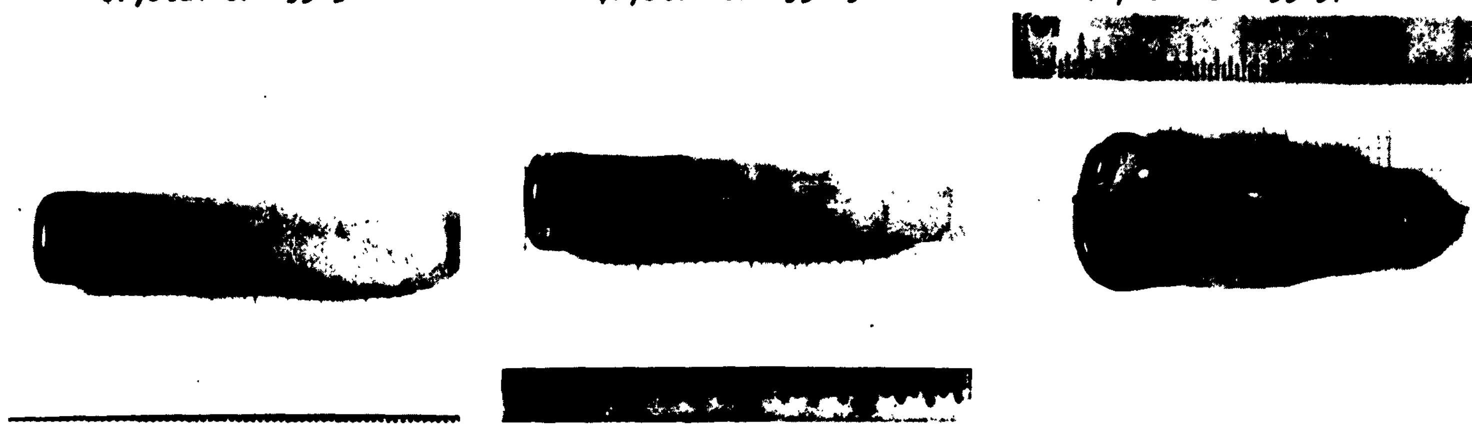


C. Crossed Polaroid

FIGURE TWO

## PHOTOGRAPHS OF CRYSTALS SHIPPED FOR LOT IV

Crystal CP-135-5 | Crystal CP-135-13 | Crystal CP-135-37



A. As Grown Crystal

B. Tyndall

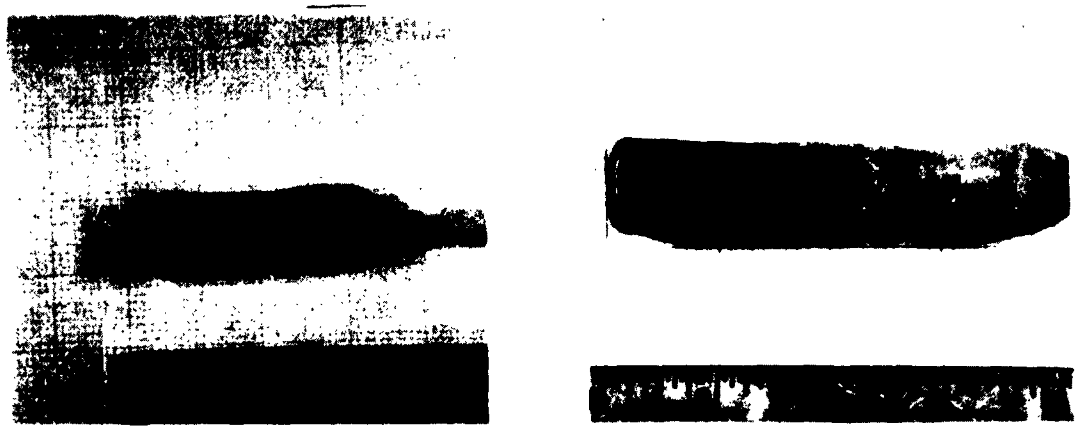
C. Crossed Polaroid



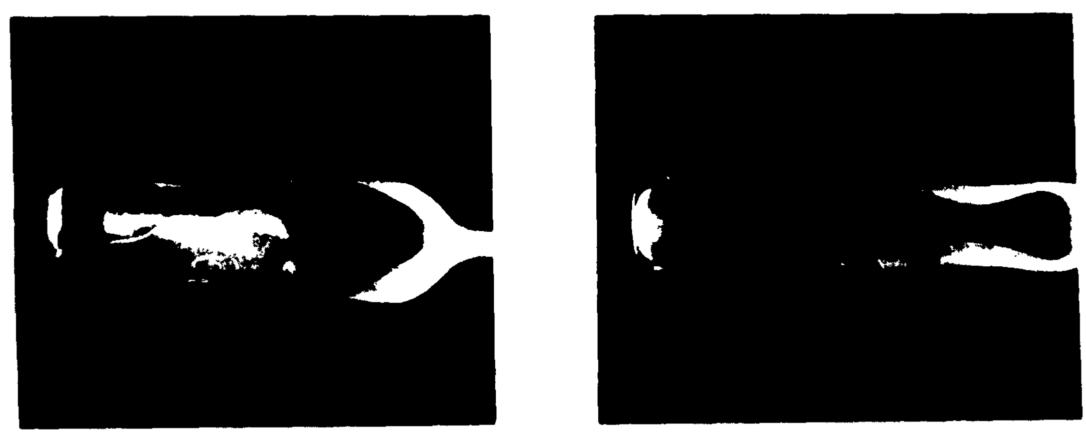
FIGURE THREE (A)

PHOTOGRAPHS OF CRYSTALS SHIPPED FOR LOT IV (CONT.)

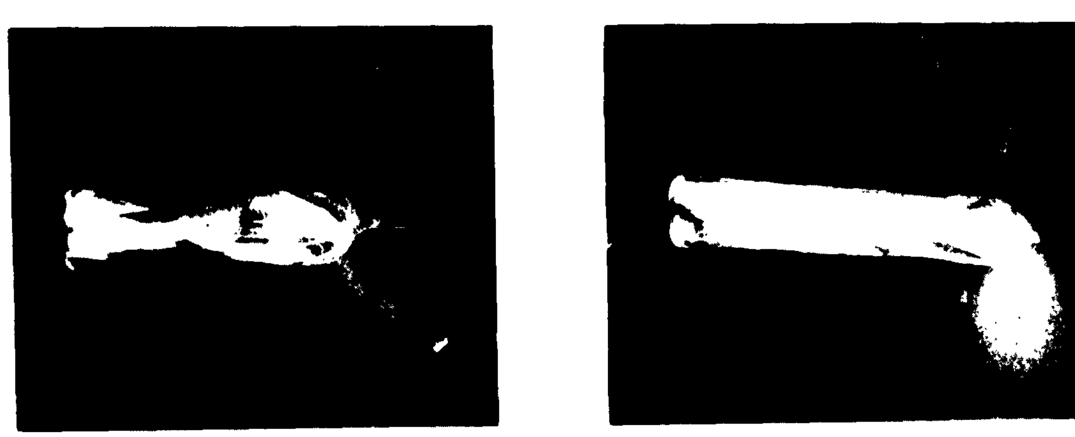
Crystal CP-135-43      Crystal CP-134-6



A. As Grown Crystal



B. Tyndall



C. Crossed Polaroid

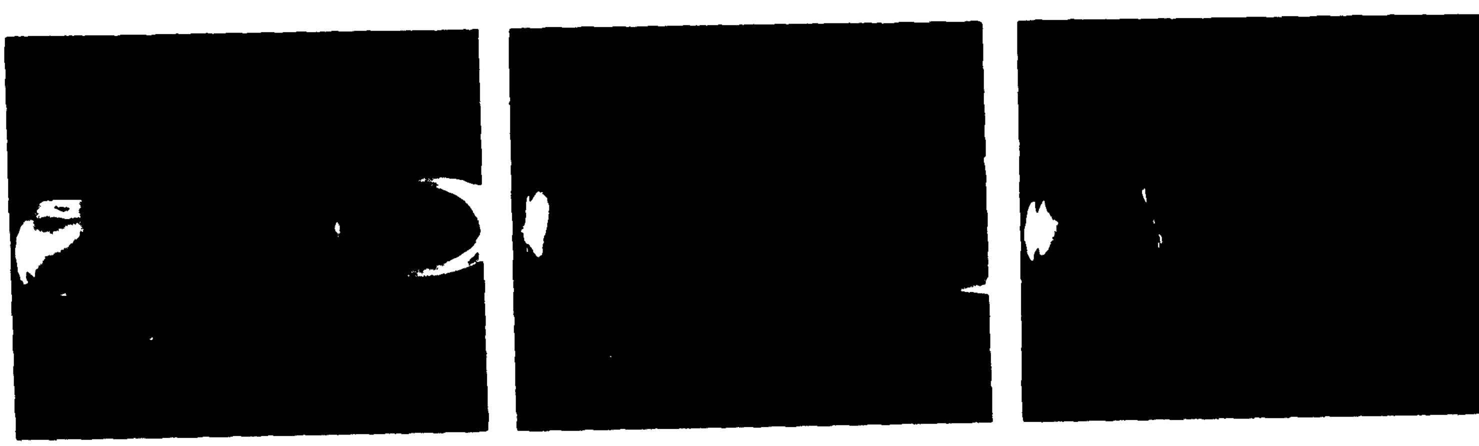


FIGURE THREE (B)

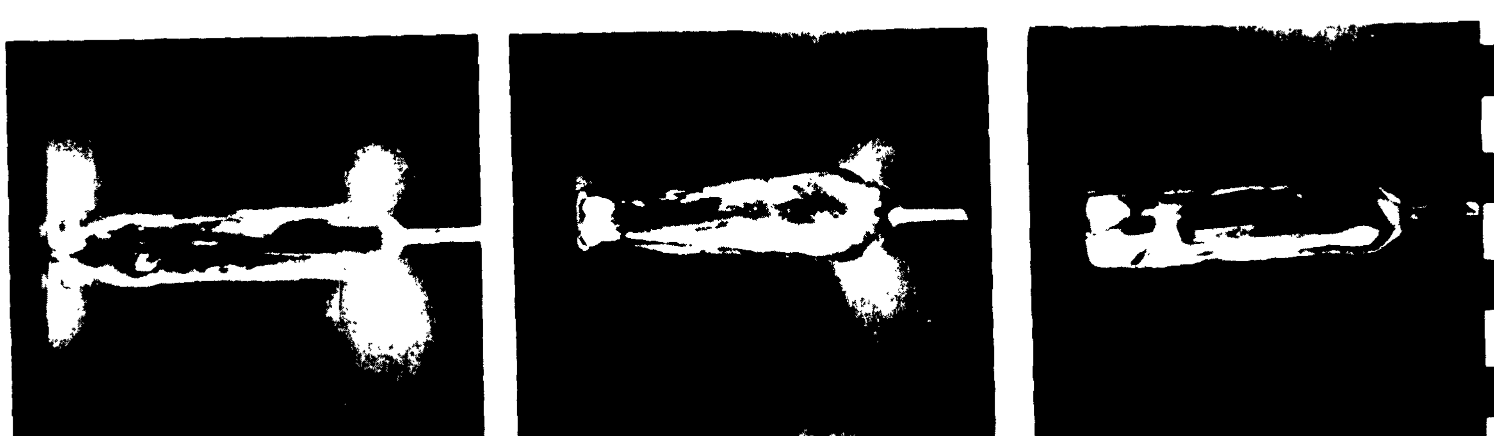
## PHOTOGRAPHS OF CRYSTALS SHIPPED FOR LOT V

Crystal CP-136-5 | Crystal CP-136-20 | Crystal CP-136-24

A. As Grown Crystal



B. Tyndall

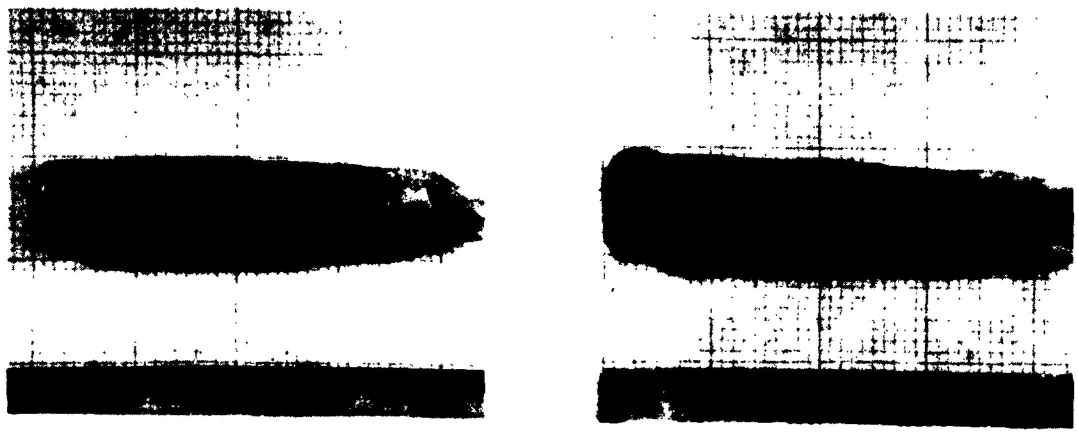


C. Crossed Polaroids

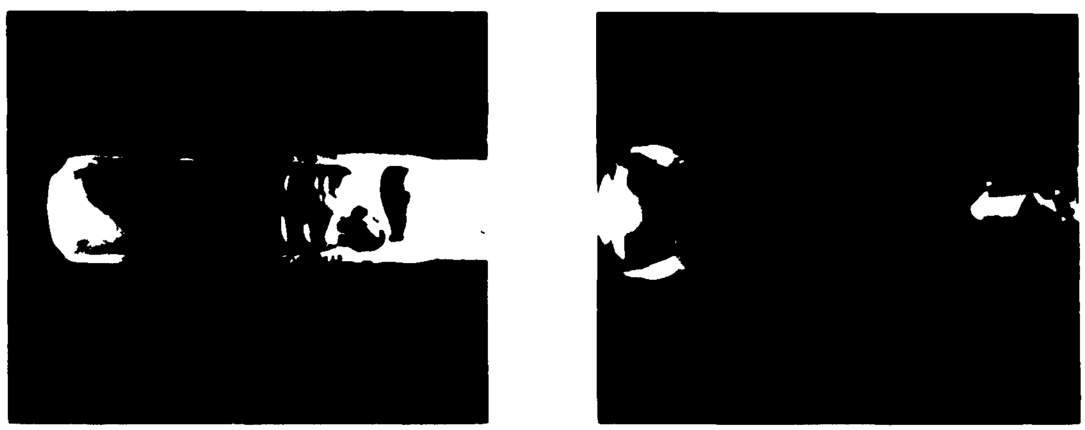
FIGURE FOUR (A)

## PHOTOGRAPHS OF CRYSTALS SHIPPED FOR LOT V (CONT.)

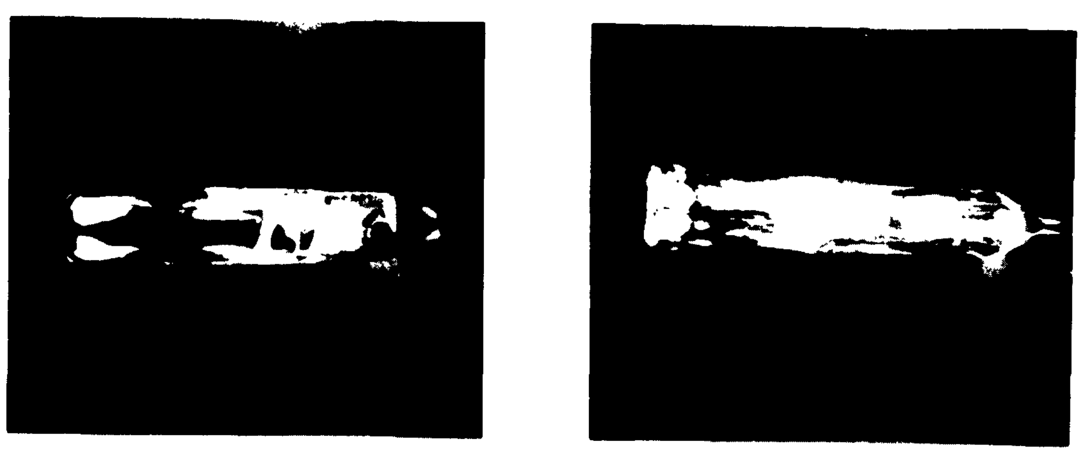
Crystal CP-136-28 | Crystal CP-136-52



A. As Grown Crystal



B. Tyndall

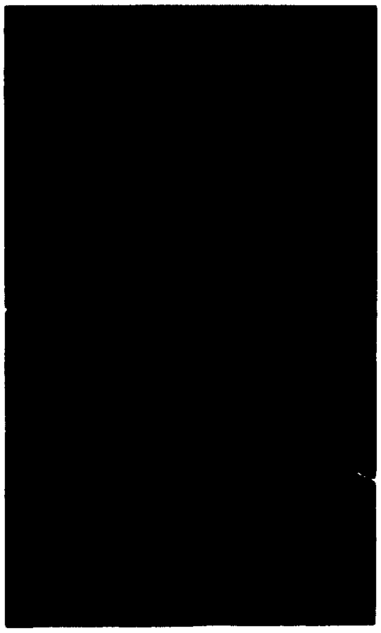


C. Crossed Polaroids

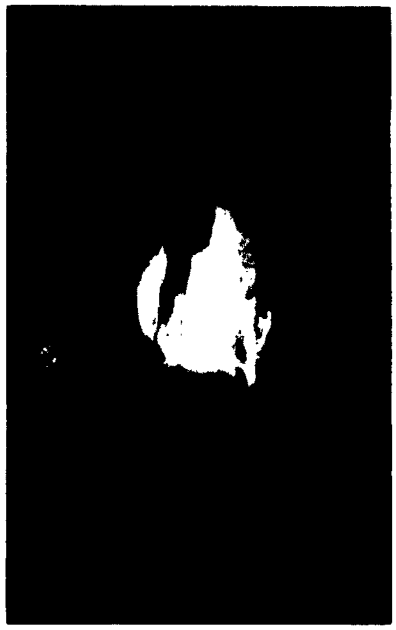


FIGURE FOUR (B)

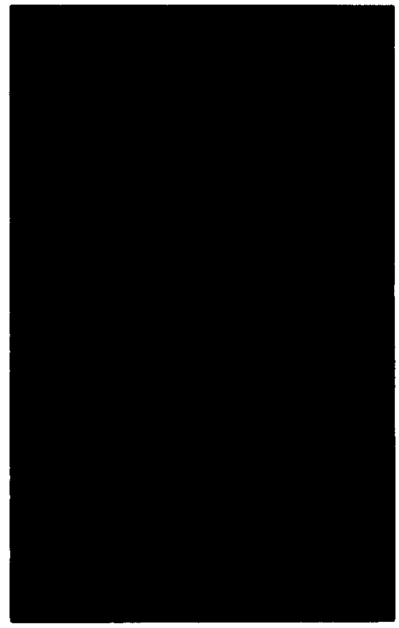
## SCHULTZ-WEI X-RAY PATTERNS FOR SEED EVALUATION



Seed #8-20-1
5' Misorientation



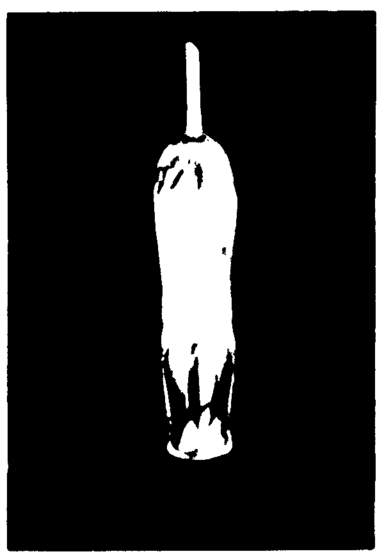
Seed #8-20-5
$2^{\circ}$ Misorientation



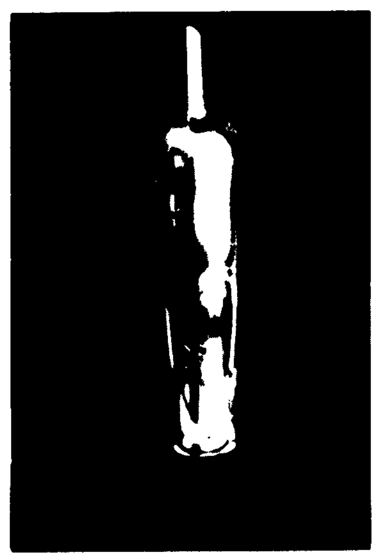
Seed 8-20-6
$1/2^{\circ}$ Misorientation

A. X-ray Patterns of Seed Caps

CP-135-37
Grown with 8-20-1



CP-136-13
Grown with 8-20-5



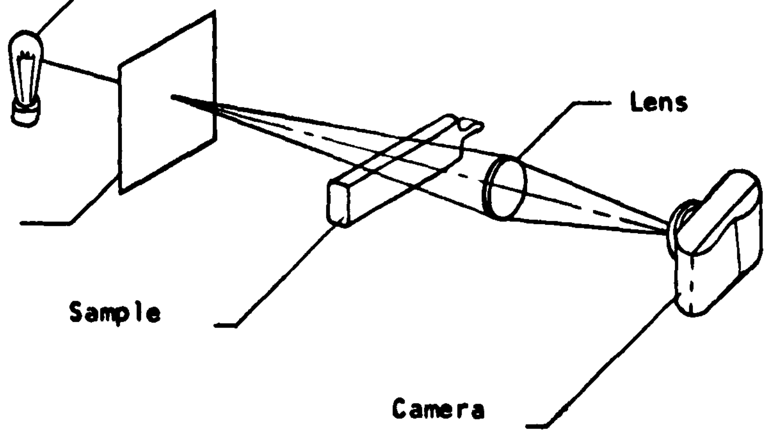
CP-136-16
Grown with 8-20-6

B. Crossed Polaroids Patterns of Crystals With Above Seeds

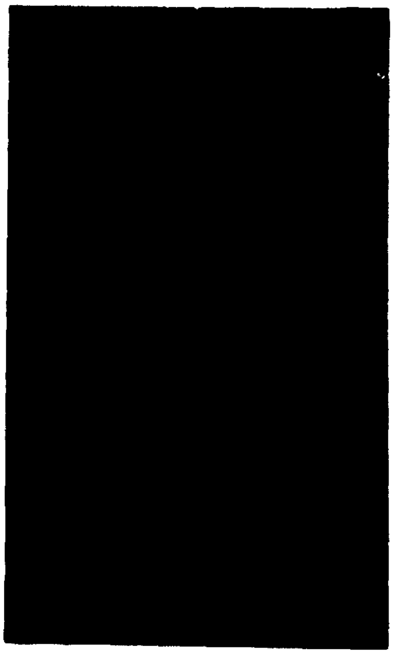


FIGURE FIVE

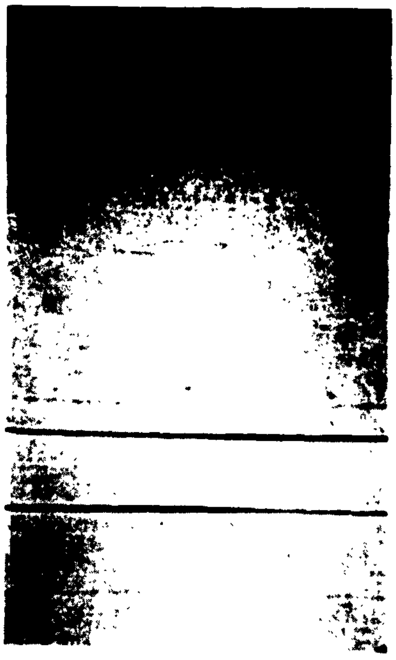
## SCHLERIN PHOTOGRAPHS OF CRYSTALS




A. Schematic of Schlerin System



CP-135-43
Uniform Light Banding



CP-134-6
Coarse Banding

B. Photographs Showing Banding



FIGURE SIX

## CONCLUSION

The following conclusions can be made on the crystals shipped under this contract to date.

1. Crystals grown with the 50°C (lot III) Thermal gradient have superior internal quality to the crystals grown with thermal gradients of 150°C (lot II) and 300°C (lot I).

2. Crystals grown with a relative growth rate of 2 (lot IV) have superior internal quality to crystals grown with relative growth rates of 3 (lot III) and 4 (lot V).

3. No evaluation of annealing cycles can be made until laser evaluation data is available.

4. Seeds used to grow crystals of high internal quality must exhibit no lineage boundaries as shown by x-ray mapping.

## PROGRAM FOR NEXT INTERVAL

The program for the third quarter (1 Nov 62 through 31 Jan 63) includes the growth of boule lot VI, required to complete the lots necessary for annealing cycle and growth rate studies; the growth of boule lot VII to study the effect of fluxing agents and the growth of boule lot VIII to study the effect of orientations other than 90°.

## PERSONNEL

| | |
|---|---|
| B. N. CALLIHAN | 18 hours |
| R. L. Hutcheson | 212 hours |

AD

Linde Co. Div. Union Carbide Corp.,
East Chicago, Indiana
RUBY IMPROVEMENT FOR LASERS-TASK I by R.L. Hutcheson, Report #2 for 1 Aug 62 to 31 Oct 62, 23p incl.illus, tables (Rept.Nr.2--2nd quarterly report) (Contract DA 36-039 SC 89089)
Unclassified Report

The effects of thermal gradients, growth rate and annealing cycles on the growth of ruby for lasers are discussed. The growth information on three lots of ruby is presented.
(over)

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1. Synthetic Ruby
2. Corundum
3. Aluminum Oxide
4. Sapphire-Synthesis
I. Title: Lasers
II. Hutcheson, R.L.
III. Army Research and Development Center, Ft.Monmouth, NJ
IV. Contract DA-36-039-SC 89089

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ABSTRACT CARDS

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